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Communication to the Editor

Determination of Chlorotoluron in Technical Products and Formulations by Reversed-Phase High Performance Liquid Chromatography (RP-HPLC)

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Abstract: An RP-HPLC method for the quantitative determination of chlorotoluron in technical products and formulations has been developed, using as column μBondapak C18, 250 × 4·6 (ID) mm, as eluent methanol + water + acetic acid (60 + 40 + 0·1 by volume), with detection by UV at 243 nm. Recoveries were 99·3–100%, RSD ($n = 5$) < 0·47%.

Key words: reversed-phase high performance liquid chromatography, chlorotoluron, herbicide, quantitation

1 INTRODUCTION

Chlorotoluron is widely used in China as an effective herbicide. For quality control there are several analytical methods available to determine the chlorotoluron content of technical products and formulations. For instance, the CIPAC–AOAC method has commonly been used in China.[1] Besides this method, there are normal-phase HPLC and GLC methods for residue analysis.[2,3]

The CIPAC–AOAC method gives reliable results for technical products and formulations of high purity, but for products having high concentrations of impurities the results are not so good. In addition, the method takes quite a long time.

In 1989, ICAMA, Beijing, developed a RP–HPLC method which has since been officially adopted by the Ministry of Chemical Industry (HG 2168-91 chlorotoluron TC and HG 2169-91 chlorotoluron WP). This method allows the separation of the impurities from technical products and formulations.

2 EXPERIMENTAL

2.1 Apparatus and chromatographic conditions

The apparatus consists of a μBondapack MT C18 stainless steel column, 250 × 4·6 (ID) mm, a Waters HPLC 6000 pump, 481 detector, U6K injector or equivalent. Column temperature: ambient; flow rate: 1·0 ml min^{-1}; injection volume 15 ml → 15 μl; retention time: *c.* 10 min.

2.2 Reagents

The required reagents are a chlorotoluron standard of known purity, methanol (HPLC grade), acetic acid (HPLC grade) and water (Millipore QII type, 18 MΩ cm).

The mobile phase is methanol + water + acetic acid (60 + 40 + 0·1 by volume), filtered through a 0·45 μm filter and degassed in vacuum before use.

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TABLE 1
Chlorotoluron Content of Technical Products (TC) and Wettable Powder Formulations (WP) Determined using RP-HPLC

	Chlorotoluron content (% m/m)							
Sample	*1*	*2*	*3*	*4*	*5*	$\bar{X}$	*s*	*CV*
TC	92·96	93·15	93·09	93·46	93·65	93·46	0·28	0·30
WP	19·24	19·31	19·23	19·07	19·17	19·20	0·09	0·47

RSD ($n = 5$) < 0·47%.

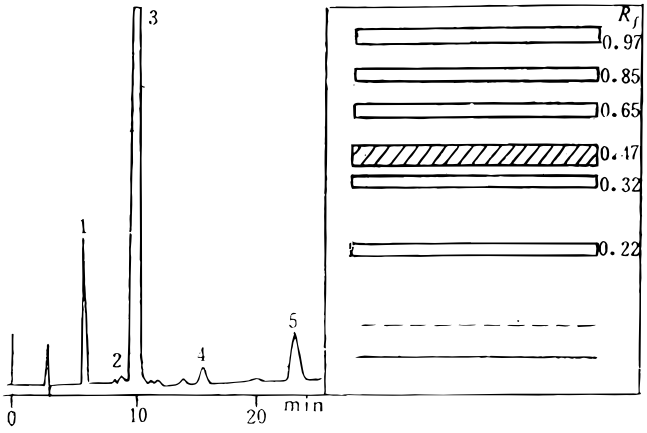

Fig. 1. Separation of chlorotoluron and impurities by RP-HPLC and TLC.

Peak(HPLC)	R_f(TLC)	Chemical name
1	0·22	3-(3-chloro-4-tolyl)-1-methylurea (by product I)
2	0·32	3-(4-tolyl)-1,1-dimethylurea(by product II)
3	0·47	chlorotoluron
4	0·85	1,3-bis-(3-chloro-4-tolyl)urea (by product III)
5	0·65	unknown

2.3 Methods

2.3.1 Standard solution

Dissolve chlorotoluron standard (0·1 g) in methanol (100 ml). Dilute 0·5, 1·0, 2·0, 3·0, 5·0 ml samples of this solution to 100 ml with methanol, to prepare standard calibration curve.

2.3.2 Test solutions

Take technical material or formulation equivalent to 0·1 g chlorotoluron and add to 100 ml methanol. Shake for 20 min. If necessary, centrifuge a portion (*c.* 5 ml) and filter using a filter syringe (0·45 μm filter) for HPLC determination.

3 RESULTS

A C-18 column is suitable to analyse chlorotoluron. The maximum UV absorption of chlorotoluron is at 243·8 nm, and the UV detector was used at 240–250 nm.

The standard calibration curve was:

$$y = 1{\cdot}91 + 51{\cdot}94x \qquad r = 0{\cdot}9999$$

Figure 1A shows a chromatogram of chlorotoluron and impurities separated from technical material by RP-HPLC and Fig. 1B shows a similar separation by TLC.

Recoveries from technical material and formulations were 99·28–100·04%. The reproducibility of the method is shown in Table 1. The method has proved to be simple, fast and very effective for determining chlorotoluron in technical products and formulations.

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